Graphs 1,2, and 3 show the result of plotting Mn (weight percent) found on the catalyst against vehicle mileage. There is considerable evidence of an increase of weight percent Mn increase with mileage. There was too much data scatter to make reasonably quantitative estimates of the rate of increase.

This is not too surprising when within the three fleets we find variables in engine sizes for each fleet and therefore in probable variations in exhaust flow rates, and in number of bricks in the systems (1 or 2). Finally, the three fleets have different mileages.

In order to reduce the number of variables, the following steps were applied:

- 1. All Mn (weight percent) values for each catalyst car in each fleet were averaged. .
- 2. Where a one brick system is used its value is divided by 2 in order to compare it with 2 brick systems.
- 3. Since the bulk of the vehicles contained 2.3 Liter engines the entire fleet was made to simulate a 2.3 Liter engine by multiplying the value of the ratio 2.3 Liters/X where X is the value in liters of the engine involved.

When these admittedly rough corrections were made linear relationships of catalyst Mn concentration versus miles were established for each fleet. This is shown in graphs 4,5, and 6. The values for these graphs were combined into graph #7.

This graph shows two very significant facts:

- 1. The rate of increase of wt% Mn is approximately constant.
- 2. Even with many unknowns a rate of the deposit can be quantitatively estimated for ceramic monolith catalysts such as are used in the Ford systems studied. For the values plotted the percent increase in weight percent of Mn is between 0.03 and 0.05 per 10,000 miles.

Question B

The next important question is whether this material lays on the surface or whether it reacts with the substrate or the washcoat. Since no specific information was available on these reactions, the

approach used was to attempt to find a "tracer" and to compare the ratio of this tracer to Mn at various locations to see if the ratios change substantially. If there was a major change in ratio a reaction would be suspected or established. If not, a reaction of the Mn₃O₄ with the rest of the catalyst would appear unlikely. Of the tracer possibilities available in the data, lead and phosphorous appeared the most useful. Graphs 8,9, and 10 represent the ratios between Mn and Pb contents in the various catalysts. A good relationship exists but there is considerable scatter. Graphs 11,12, and 13 show less scatter for Phosphorous so this was used in the ratio comparison. Table 1 shows the ratios of Mn weight percentage to P weight Percentage multiplied by 10 for convenience. This table covers the 9 car fleet. Table 2 shows the ratios for the 26 catalyst No statistical analysis of the ratios was performed because observation shows that any variation in the ratios is within the analytical accuracy of the methods used. Variations in the ratio occur randomly in the front middle or back of the first brick where Mn concentrations are highest. they also occur randomly in the front middle or back of the second brick where Mn concentrations are lower. This established that the Mn_2O_4 , a very stable compound, does not take part in any reactions.

Question C

The third question to be answered is: Are the deposits truly porous or do they restrict the exhaust gas reactions that should occur in the catalyst?

Extensive surface area measurements were made by Ford using the B.E.T. surface area technique. It is reasonable to conclude that if the $\rm Mn_3O_4$ is not porous, there should be a relationship between $\rm Mn_3O_4$ and B.E.T. values. In other words, the higher the Mn weight percentage values, the lower the B.E.T. should be. Chart 14 is a comparison of all the points available in the reports. There are many points since Mn and B.E.T. were taken on the brick slices. In this case the profusion of data is not very enlightening as far as establishing a relationship is concerned. In fact a lack of correlation is shown.

The various groups of data were then analyzed. Graphs 15, 16, and 17 show the result. Again there was no correlation but an interesting fact emerged. The 11 Car group showed exceptionally high B.E.T. surface areas. The observations on Page 4 of Ford Attachment 2 led me to draw a line at B.E.T. values of 5.0 M²/g to indicate an unacceptable catalyst as defined by the ford investigators².

Graphs 18, 19, and 20 show the relations between mileage and B.E.T. They bring no enlightenment on the cause or catalyst deterioration. It is clear from those graphs Mn₃O₄ does not cause macro or micro plugging which affects catalyst reactions at the Canadian concentrations of 1/16 gram of Manganese per gallon. Since there is no correlation between B.E.T. area and Mn concentration.

Based on the foregoing data it seems that:

- 1. The Mn₃O₄ formed, apparently at some point prior to the catalyst, slowly forms deposits on the catalyst at a constant rate.
- 2. There is no indication, however, that Mn_3O_4 enters into any chemical reactions within the catalyst.
- 3. The Mn₃O₄ coating is apparently porous, and does not appear to interfere with the area on which the chemical reactions of exhaust pollutants occur.

If Mn₃O₄ does not cause the reduction of chemical activity, what does?

CHEMICAL REACTIONS INCLUDING THOSE OF Mn, WHICH COULD AFFECT CATALYSTS.

Perhaps the best place to start is with the full quote of the paragraph on page 4 of Ford attachment 2 referenced earlier.

X-ray diffraction analysis of samples from vehicles with accumulated mileage in the ranges of 10K to 40K, 21K to 30K, 31K to 40K, and 41K to 50K showed that samples

#107, #108, and #109 had the α -alumina phase present. This α -phase indicates that these catalyst had undergone exposure to temperatures >1000° C at some time during their operation. Sample #112 from the 31-41K mileage range did not show the α -alumina phase present. X-Ray diffraction analysis also confirms the rust colored residue to be Mn₃O₄.

B.E.T. surface activity measurements on the second series (Table 3) range between 1.25 and 22.56 m²/g for the first brick and between 0.61 and 21.61 m²/g for the second brick. Catalysts #100, #108, and #110 have B.E.T. values below 5 m²/g which indicates their exposure to operating temperatures greater than 1000° C. These values confirm the XRD results. Catalyst #103 had only one brick which had been abraded to the size of a baseball but still had a B.E.T. value of 9 m²/g. In comparison, in-use vehicles having BET's greater than 5 m²/g are considered acceptable and BET's for fresh catalyst fall in the range of 25 m²/g.

The underlining has been added to emphasize certain points.

The reactions in a catalytic converter are, generally, strongly exothermic, since both carbon monoxide and hyrocarbons give off considerable heat in their oxidation as can be seen by consulting any standard reference. Actually NO formation requires a small amount of heat. As can be seen from reading exhaust analyses reported by various authors it is apparent that the HC contents of exhaust gas are small compared to the CO content, therefore the temperature of the exhaust gas is dependent on: inlet temperature to the reactor and combustion of percent CO X 155° F temperature loss through the reactor.

The derivation of the factor of 155° F per percent CO combusted is described in SAE paper 486J³. The "reactor" could be the manifold as is the case with thermal reactors, the catalyst or even the engine itself.

We will not discuss engine exhaust temperatures, but starting with the engine exhaust there will be some additional clean-up in the muffler if the time and temperature conditions are right. SAE paper 486G⁴ deals with these reactions. According to this reference a minimum of 1200° F is required to initiate this reaction, and about 4% CO is required to maintain it.

SAE paper 486J describes the thermal conditions for 1.5% by volume of CO (the then existing California standards) and shows that they ranged on the average from 950° F at the manifold to 700° F for catalytic muffler inlets 100 inches removed from the manifold outlets. This is in good agreement with data presented in SAE paper 760781⁵ where exhaust outlet compositions average at least 1% CO less so that about 155° F should be removed from these values. In other words about 800° F to 650° F should be the current inlet temperatures. Experience has shown that average catalyst intake temperatures range around 650° F. this then is the gas with which the catalytic converter operates.

Catalysts of the types described usually consist of a ceramic substrate of Cordierite (4(Mg,Fe) $0.4~\text{Al}_2\text{O}_3$ $^{\circ}$ $10~\text{SiO}_2$ $^{\circ}$ $\text{H}_2\text{O})$. The surface area is insufficient so a "wash-coat" of Gamma alumina is washed over and through the Cordierite to provide very large surface areas of the order of 100-200 square meters per gram. The precious metal catalyst is distributed throughout the wash coat and has general access to the exhaust gases.

For thermodynamic reasons nature abhors large surface areas and under various conditions will move to decrease them. Well known examples of this phenomenon are grain growth in metallurgy and crystal growth in solutions. Gamma Alumina is no exception. At temperatures ranging somewhere from 950° F to 1800° F (1000° C) the gamma alumina converts to alpha alumina which has a much lower surface area. The extent of conversion depends on temperature and time at this temperature. The catalytic effect depends on the rate of reaction.

Figure 21 compares the average B.E.T. values with light-off temperature values derived from graphs submitted by Ford for the

11-Catalyst Fleet and the 26-Catalyst Fleet. The light-off point was an estimate which attempted to show where a change in the rising concentration occurred. Since each of the curves in the 26-Catalyst fleet had different shapes the points could vary somewhat. The values at the light-off point for the HC and NO conversion are shown under the estimated "light-off" point for the 26-Catalyst Fleet and one doubtful point on the 11-Catalyst fleet. Other conversion values were not shown for the 11 Catalyst fleet because all were about 80% for HC and 90%+ for NO.

None of the CO points were recorded, since they were in the 90%+ range for both fleets at the light-off points. Figure 21 shows that the vehicles from the 11-Catalyst Fleet have little loss in reactivity or surface area, whereas both the 9-Catalyst Fleet and the 26-Catalyst Fleet show extensive loss in surface area and reactivity. This is presumably because of over-temperature conditions since it has been established that MMT is not a factor in the loss of surface area.

From previous data cited above it can be concluded that the catalysts were operating in CO concentrations of 2-6% for considerable periods of time to accomplish the temperature deterioration shown.

The MECA submissions on page 3 shows some concerns about reaction with the cordierite which occur at temperatures ranging from 1475° F to 2456° F (800° C to 1347° C). These appear to be technically interesting but also to be non sequiturs because of the fact that the active surface of the catalyst would have been fatally altered before these temperatures were reached.

The question that arises from all of this, is what caused the excessive temperatures? Was there some other cause of catalyst failure? There can be no serious suggestion that abnormal catalysts were at fault for the catastrophic results reported by Ford. First of all, there was no general pattern of distribution of the area of operation and no indication that they were all from the same time of manufacture. Furthermore, the 11 Catalyst Fleet and the Ethyl Waiver Fleet along with extensive other testing has demonstrated consistently good performance when all of the factors that affect

engine and engine controls performance are according to specification. This includes particularly the oxygen sensors.

Unfortunately, the oxygen sensors for the 26 Catalyst and 9 Catalyst Fleets were not examined. They were not available for the 26 Catalyst Fleet which is probably the case with the 9 Catalyst fleet. The sensors for the 11 Catalyst Fleet were sent to Robert Bosch Co. for detailed analysis. According to Bosch all were within specification for functioning oxygen sensors, but two showed "abnormal behavior" (unspecified type). They were from vehicles BK7 and BK9. BK7 also showed a loss in B.E.T. level. Two questions arise:

- 1. Is the "abnormal behavior" in any way related to MMT? (Probably not in view of the excellent performance of oxygen sensors in the Ethyl Waiver Fleet.)
- 2. Are the specification limits for what constitutes a functioning oxygen sensor too broad?

Detailed clarification of these two questions from Robert Bosch Co. would certainly be helpful.

Similar questions about field failures of oxygen sensors should be asked of Bosch and the automobile manufacturer. Finally, MECA should be asked to elaborate on "undesirable reactions" which concern their "catalyst engineers"?

I wish to express my thanks to the various submittors, and particularly to the Ford Motor Company for their extensive and detailed evaluation of field vehicles. Their data made it possible to answer the MECA concerns scientifically rather than add more oratory to the docket.

The Future

The speculation about future standards is difficult to address because they have been in the discussion phase for about ten years.

There are two possible effects on the exhaust reaction conditions that may result from lowering present standards:

1. The catalysts may be coupled directly to the exhaust manifold. Since catalysts are currently coupled as closely as 8 inches from the manifold, temperature increases of not more than 25° F could be experienced, based on the temperature reductions along the exhaust system shown in the references cited. This is well within the variations in temperature that will occur as a result of operational variables such as ambient temperature or traffic conditions.

2. As cited previously, the only other theoretical effect would be from more combustion in the catalyst as a result of lowering the standards. As has been pointed out the only important factor in raising catalyst temperature is the CO combustion. Since well over 95% conversion of this compound is currently being accomplished the difference of a couple of percentage points is all that we are talking about. Converted to catalyst temperatures this will not be more than 5° F.

Thus, it is quite obvious that no substantial increase in catalyst temperature can be expected. In a way, this in unfortunate, because higher temperatures promote higher conversion rates. Even such desperation moves as heated catalysts have been proposed, but when you are at 95% or more conversion additional improvement becomes very difficult to accomplish or even measure.

I expect that further improvement in the engine output as a result of modifications of the injection systems and the oxygen sensors and other engine components will result in cleaner exhaust stream to the catalysts. The catalysts themselves will probably be improved as a result of continuing production control efforts. The combination will possibly result in meeting some of the more reasonable proposed reductions.

At this point the evaluation techniques will probably need to be revised. New traffic cycle studies will have to be made since the ones on which present evaluations are made are 25 years old and undoubtedly outmoded. New instrumentation may need to be developed. Even though the atmosphere may only experience a 5% reduction or less in pollutants as a result of a proposed new

regulation, the instrumentation will be asked to analyze a 50% reduction of the concentration in tailpipe emissions that it is presently analyzing.

Atmospheric measurements are currently incapable of evaluating he major changes that have been made, so a large program in this area is called for.

These seem like huge programs, and they are, but the new standards proposed involve expenditures of billions of dollars per year, and these steps will be required to see if the American public is getting its money's worth.

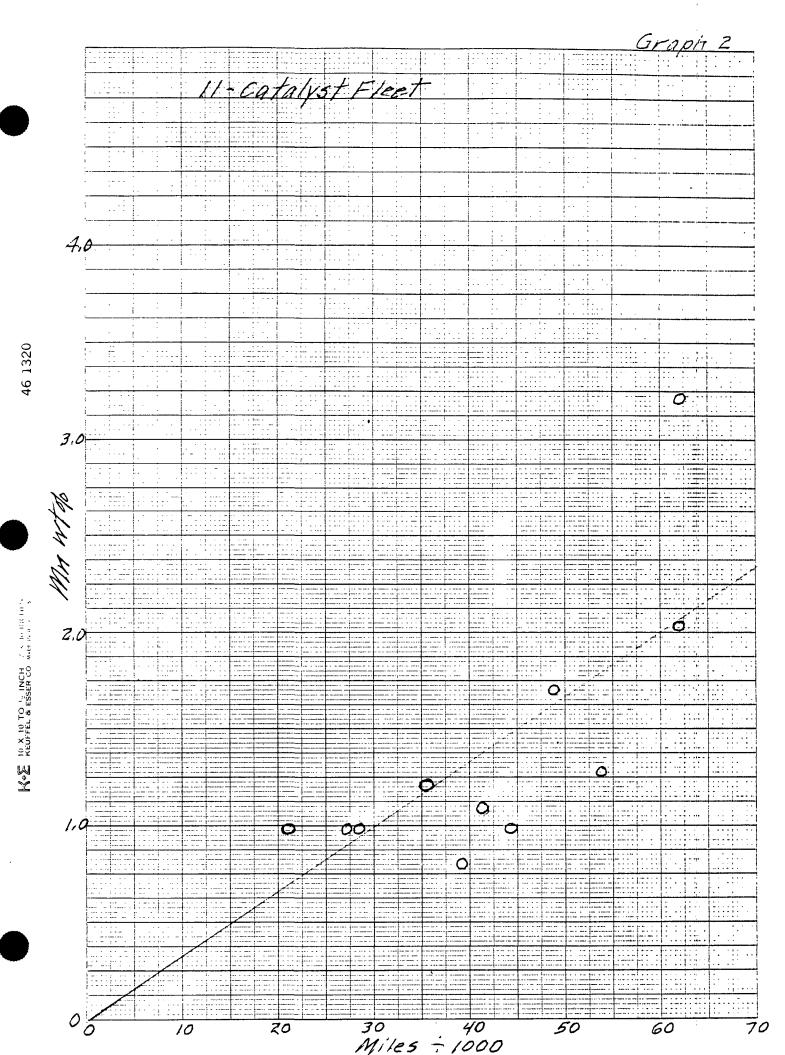
These then are some of my speculations on the future. Consulting Nostradamus may be more productive.

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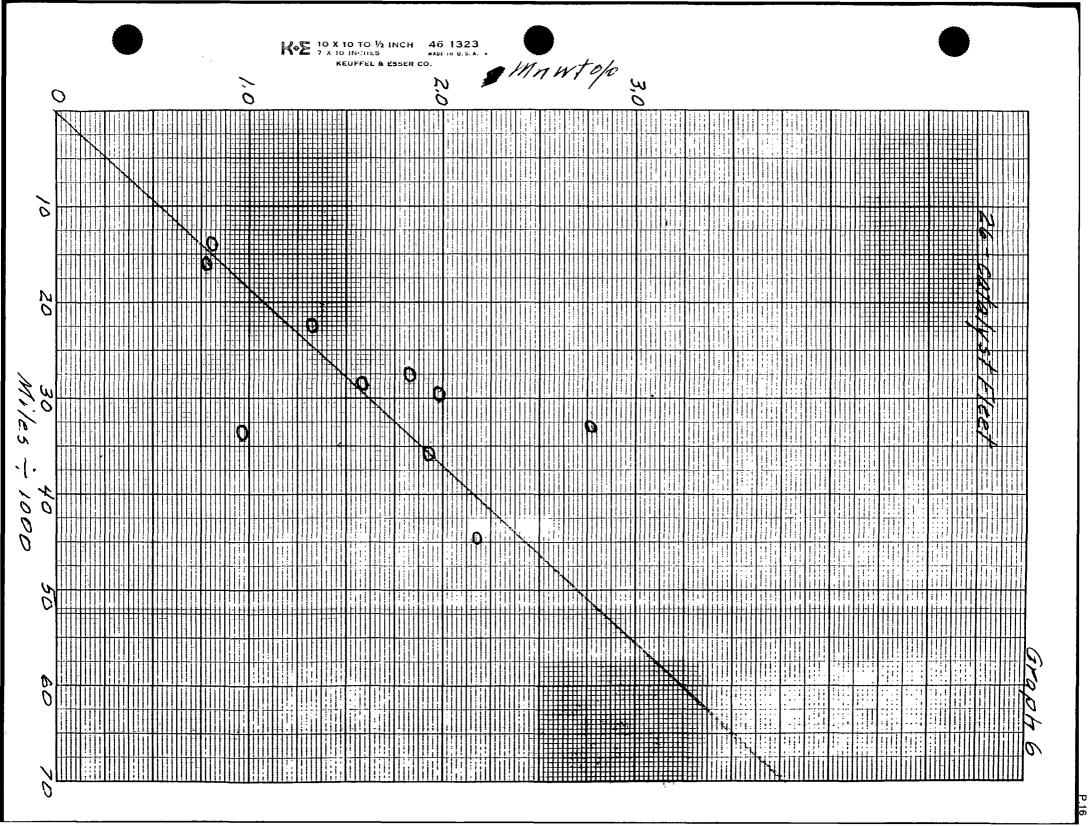
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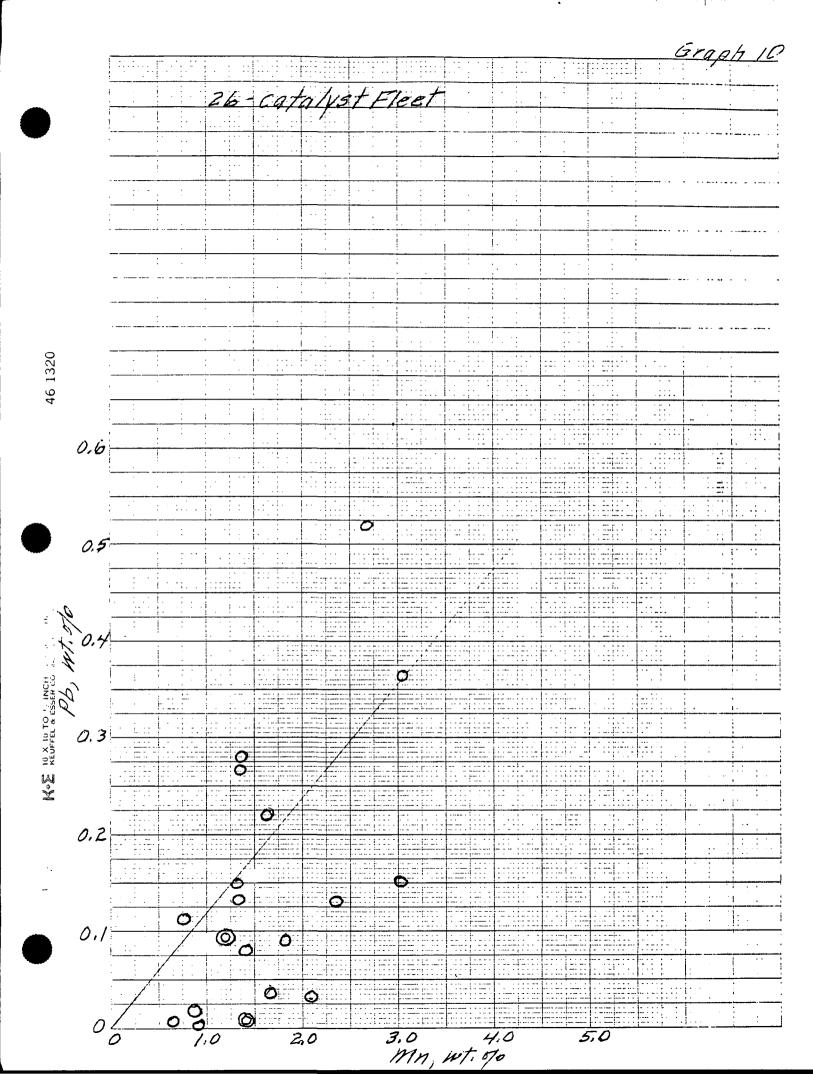
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<u>P.35</u>

JUMMARY OF 26 CATALYST FLEET- P3

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<u>C</u>	0.04	0.08	6.38 0.20 0.74	0.12 0,12 0.08	6,09 0.02 0.67	0.05 6.04 0.02	
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Soichiro Honda Medal

CHARLES M. HEINEN

Conferral at the Honors Assembly, 1990 Winter Annual Meeting

THE SOICHIRO HONDA MEDAL recognizes an individual for an outstanding achievement or a series of significant engineering contributions in developing improvements in the field of personal transportation. This medal was established in 1983 in recognition of Soichiro Honda's exemplary achievements in the field of personal transportation.

CHARLES M. HEINEN, retired, Bloomfield Hills, Michigan, for his leadership and outstanding promotion of automotive air pollution control activities in the automotive industry.

Mr. Heinen retired from Chrysler Corporation as director of research and materials engineering after more than 40 years with the company. Following his retirement, he consulted for several years as director of the Automotive . Research Group.

He began his career with Chrysler in 1934 as a stock handler while also attending night school at the Chrysler Institute. Then from 1938 to 1942, he attended the University of Michigan, Ann Arbor, as a Walter P. Chrysler Scholar where he earned B.S. and M.S. degrees in chemical engineering.

Following graduation, he worked on the Manhattan Project as a laboratory

supervisor with the Chrysler Atomic Project until 1945. For the next several years he was active in several materials fields, with particular emphasis on fuels and lubricants. Later, as a materials engineer, he was responsible for reclamation and waste disposal.

In the 1950s studies of apparently clear vehicle exhaust revealed possible environmental problems. Mr. Heinen's work for Chrysler in this area led to combustion studies, control devices, fuel economy efforts in light materials and power rating devices, alternate engines such as turbines, electric motors, and vehicles powered with hydrogen and alcohol. Nearly 100 of the 130 papers he presented in his career were as a result of his work in this field. He continued to work in this area as he rose from director of vehicle emissions planning to director of emissions/fuel economy and materials engineering and finally to director of research and materials engineering in 1978.

He was named a Fellow of the Society of Automotive Engineers (SAE) in 1980 for his outstanding contributions in the field of automotive air-pollution control and of the American Society of Metals (ASM) in 1977 for his distinguished contributions in the field of metals and materials.

Mr. Heinen has served as director of the Coordinating Research Council (CRC) and the Air Pollution Control Association, and he is also past chairman of the CRC Group on Combustion of Exhaust Gas, the SAE's Fuels and Lubricants Activity Committee and Engineering Material Council, and the Motor Vehicle Manufacturers Association of the United States' Air Quality Committee. He is a member of the U.S. Chamber of Commerce Committee on the Environment, the ASM Committee on Government and Public Affairs, and member and chairman of

numerous committees dealing with fuels, lubricants, and standardization for the American Society for Testing and Materials.